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SPRAY DEPOSITED NI DOPED ZNO NANOPARTICLES FOR AMMONIA VAPOUR SENSING

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ABSTRACT

ZnO is one of the most important semiconductor materials having a variety of applications in photonic, field emission and sensing devices. Ni doped ZnO films were deposited on glass substrate by spray pyrolysis technique. X-ray diffraction result indicates polycrystalline nature of the films with hexagonal wurtzite structure. The SEM and AFM micrographs indicate the presence of nanocrystallites with agglomeration present over the film surface. The grain size is found to be 15-95 nm. The prepared ZnO doped films exhibit direct band gap.. The vapour sensing characteristics of the Ni (0.0002M) doped ZnO (0.1) film were studied by chemiresistive method for various concentrations of ammonia vapour at room temperature (~ 30 °C). The gas sensing studies indicate that as concentration of ammonia increases the sensitivity increases. For 80 ppm of ammonia vapour, the film shows a response and recovery time of 12 and 21 s respectively.

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INTRODUCTION

Thin films can be prepared by many methods, and each method has its own characteristic merits and defects in producing homogeneous and defect free thin films.

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Several techniques available to form a thin film such as molecular beam epitaxy (MBE)[1], radio frequency sputtering (RF)[2], thermal evaporation, successive ionic layer adsorption and reaction method (SILAR)[3], chemical vapour deposition (CBD)[4], Sol-gel[5] and d.c magnetron sputtering[6]. Among many methods available, spray pyrolysis technique is chosen for this study due to its simple, inexpensive and reproducible quality. It is well known that the sensing performance of gas sensors can also be enhanced by the doping of noble metals (Au, Pt, Pd, etc.) or other semiconductors and so on[7-10]. Shampa Mondal et al.,[11] have reported that Al-doped ZnO thin film gas sensor shows better gas sensing performances to LPG gas compared with the undoped sample. R. Ferro et al.,[12] have investigated the Cd-doped ZnO film for NO_2 vapour sensor application. M. B. Rahmani et al.,[13] have explained about the transition from n- to p-Type of Spray Pyrolysis Deposited Cu Doped ZnO Thin Films for NO_2 sensing application.

The goal of this study is to deposit Ni-doped ZnO thin film via the spray pyrolysis technique and to investigate the structural, optical, morphological properties of pure and Ni doped ZnO thin film and to study the NH_3 sensing properties.

EXPERIMENTAL TECHNIQUE

The spray pyrolysis setup consists of a spray gun with carrier gas flow tube and a solution tube with a narrow nozzle. This carrier gas flow produces solution spray. Purified air is used as a carrier gas. The distance between the nozzle and the substrate is kept at 30 cm and sprayed at an angle of 45^o. To avoid thermal shock due to excess cooling, the solution flow rate is maintained at 3 ml/min. The spraying is done for one second and a 2 minute waiting period is allowed between successive spraying, to avoid excessive cooling of the substrate. After each application of the spray, the substrate is rotated through 90^o to make uniform coating. The temperature of the substrate is maintained at $\pm 2^oC$ through a Microprocessor based temperature controller and measured by a Chromel-Alumel thermocouple[14].

Nickel doped zinc oxide thin films were prepared from the precursor solution by dissolving the salt of Zinc acetate ($Zn (CH_3COO)_2.2H_2O$) of 0.1M and nickel (II) acetate tetra hydrate ($Ni(CH_3COO)_2.4H_2O$) of 0.001M in deionised water. The aqueous solution was then sprayed on the preheated glass substrate kept at 230^oC. Compressed dry air at a pressure of 12 Kg/Cm^2 from an air compressor via an air filter-cum regulator was used as the carrier gas and spray rate of the solution was maintained at 3 ml/min. Films were all prepared from 0.002 to 0.004M of precursor salt (Ni) without changing other parameters.

Structure of the films was investigated with X-ray diffraction (XRD) with Cu Kα radiation (1.5406˚A) using a Rigaku diffractometer. Field emission scanning electron microscopy (SEM) images of the samples were taken with a JSM-6500F. The topography of the film surface was observed using atomic force microscope (AFM, Park System, XE-170). For optical characterization, absorption and transmittance was measured using a UV-VIS-(Jasco-V-570) spectrometer in the wavelength range from 300 to 1200 nm. The sensing properties were carried by using a computer controlled National Instruments–data acquisition board (NI-DAQ 6212) interfaced with LabVIEW software to record voltage and then, to convert into electrical resistance of the film.

RESULT AND DISCUSSION

3.1 Structural Investigation

A current investigation of doping effects on the properties of ZnO films was studied by using several techniques. Fig.2 represents the XRD patterns of the undoped and NiZO films of various doping concentrations. From the diffraction patterns, both undoped and Ni doped ZnO films have the diffraction angles at 32^o, 34^o and 36^o for (101), (001) and (002) respectively. It is confirmed that the all samples exhibit hexagonal wurtzite structure with polycrystalline in nature. Among these peaks, (101) is the maximum intensity peak indicating a very high crystalline quality and C-axis growth for all samples [15]. No other impurities or related to NiO_2 is observed from the XRD peaks, indicating that effect of doping did not change the crystal structure of the ZnO film.

The maximum intensity diffraction peaks at 36^o for 101 plane obtained from the fig. The texture co-efficient was calculated for different peaks by using the following formula[15]

$$TC(hkl) = \frac{I(hkl)/I_r(hkl)}{[(1/n)\sum I(hkl/I_r(hkl))]} \text{------------------}1$$

Where I (hkl) is intensity of the film obtained XRD data, n is the number of peaks detected and Ir(hkl) is the standard diffraction pattern (JCPDS data card no(36-1451).The three diffraction peaks used to calculate the texture coefficient namely, (101), (002) and (111). From that calculations, it is clearly observed that the films growth in preferred orientation along c axis. The texture coefficient of undoped and Ni doped films are given in the Table. These results indicate that the doping concentration affects the growth orientation of the film.

The lattice parameters a and c values are calculated by using the following expression[16].

$$a = \sqrt{\frac{1}{3}}\frac{\lambda}{\sin\theta} \text{------------------}2 \qquad c = \frac{\lambda}{\sin\theta} \text{-------------------------------}3$$

This calculated lattice parameter values are in close agreement with standard values (JCPDS). The grain size of undoped and Ni doped films were calculated by using the well known Debye Scherer formula[17].

$$D = \frac{k\lambda}{\beta\cos\theta} \text{---------------------------}4$$

where K is the shape factor (0.9), λ is the wavelength of Cu Kα, β is the full width at half maximum of the highest intensity peak. On increasing the doping concentration, increase in grain was observed. (Tab).These results are well in accordance with the previously reported results[18]. The strain value was calculated by the following relation[19].

$$\varepsilon = \frac{\beta\cos\theta}{4} \text{-----------------------------}5$$

Strain is defined as mismatch between film and substrate. From the table, the low strain value (-4.045%) is obtained for (0.0002M) Ni doped (0.1M) ZnO film.

$$\sigma = \frac{2c^2{}_{13} - c_{33}(c_{11}+c_{12})}{2c_{13}}\frac{c-c_0}{c_0} \text{---------------------}6$$

where c_{ij} represent elastic stiffness constants for ZnO single crystals; c_{11} = 208.8 GPa, c_{33} = 213.8 GPa, c_{12} = 119.7 GPa, and c_{13} = 104.2 GPa [20]. The calculated stress value stress value obtained by the above formula. The positive (tensile) or negative (compressive) of the stress value could be absorbed in the undoped and Ni doped samples and these parameters are listed in the table. The less defect obtained for the Ni (0.0002) ZnO (0.1) film, which is used to detect the ammonia vapour and suitable for optoelectronic application.

3.2 Optical Study

The optical absorbance and transmittance of undoped and Ni-doped films were recorded in the wavelength range of 350-1100 nm. As can be seen from the fig 5, with increasing Ni concentration, the absorbance of the films increased. In fig.6,a decrease in optical transmittance with increasing doping concentration is observed. It is seen that the film with lower Ni concentration has maximum transmittance and the films exhibit the high average transmittance of 70% at visible region and absorption edges shift towards the lower wavelength region as Ni concentration is increased.[21]

The optical band gap of the film is determined by using the following formula[22]

$$\alpha h\nu = B\sqrt{h\nu - E_g} \text{ --------------------------------}(7)$$

where α is the absorption coefficient, hν is the photon energy, Eg is the optical band gap and B is constant. The optical band gap were determined from the Tauc plot of $(\alpha h\nu)^2$ versus hν. The optical band gap of the films is in between in the range of 3.27 eV to 3.44 eV with the lowest value for NiZO-2 (3.27 eV) and highest value for NiZO-1 (3.44 eV). The lowest optical band gap along with good transmittance for NiZO-2 sample can be employed the application of vapour sensor detection.

3.3 Topographical Properties

The topographical investigation of pure and Ni doped ZnO films deposited on glass substrate at 230°C was done using AFM. Typical 3D AFM images of the both films synthesized at different molar concentrations are shown in Fig. 5 (a) and (b). AFM results showed the homogeneity of the films and have fine pores were observed in some places. The root mean square (RMS) roughness for undoped and Ni doped ZnO films were 12 and 16.4 nm from AFM respectively.

Doped film has increased crystallite size as well as increased roughness than the undoped film. The increase in crystallite size may be caused by columnar grain growth in the structure[23]. The results of arrangement of particles obtained from AFM images are in good agreement with those obtained from XRD measurements shown in fig.1.

3.4 Morphological Studies

The morphology of undoped and Ni doped ZnO films are given in fig.6. (a) and (b). As can be observed from the figure, both films show agglomeration and particles are present in some places. The Ni doped film is more closely packed and is porous in nature compared to pure ZnO film. These results are in confirmation with AFM images (fig. 6.(a) and (b))

3.5 NH_3 Sensing Properties

The sensing properties of Ni-doped ZnO thin film towards the detection of NH_3 vapor in the range of 20-140 ppm at room temperature was investigated. The fig. 8 reveals the decrease in resistance of the film when ammonia was injected and the flow of air was stopped.

The gas sensing mechanism of Ni-doped ZnO film is mainly attributed to the change of resistance caused by the adsorption and desorption of ammonia molecules on the surface on the film. When air is exposed to the film (0.0002M)Ni(0.1M)-ZnO, the surface will absorb the oxygen molecules, which in turn get converted to oxygen ions capturing electrons from the conduction band of Ni-ZnO. As a result, the resistance of the film will increase due to decrease of carrier concentration. The schematic diagram of sensing mechanism is given in fig.7.

When the film (0.0002M)Ni(0.1M)-ZnO is exposed to target gas like NH_3, the ammonia molecules react with adsorbed oxygen ions on the surface of the film and the gained electrons are released to the conduction band of the Ni-doped ZnO film resulting in the reduction in resistance of the film.

$$2NH_3 + 3O^-(ads) \longrightarrow N_2 \uparrow + 3H_2O \uparrow + 3e^- \downarrow in\ resistance \text{ ---------(8)}$$

The gas response of the sensor was obtained using the relation[24]

$$\text{resistance change} = \left| \Delta Rair - Rgas \right| \text{ ----------------(9)}$$

It is observed that the change in resistance increased with gas concentration[25]. , where Rair the resistance of the sensor in air and Rgas is resistance in target gas and the calculated value is given in the Table. 3.

3.5.1. Response and Recovery Studies

The response and recovery times of N- doped ZnO thin film exposed to ammonia vapour is given in the Table.3. In comparison with the sensing behaviour of the undoped ZnO thin film exposed to ammonia studied by the same authors, it is observed that the doped film exhibited quick response and faster recovery time. This can be attributed to the presence of Ni ions, which reduces the defect in film as seen from the reduction in strain in doped film compared to the undoped ZnO film[26].Also due to the influence of crystallinity, which is related to the grain size and calculated value is given in Table.2. The calculated defect values from XRD data is given in the Table 2. Moreover, the quick response (12 sec) and recovery (21 sec) times were obtained at 80 ppm is given in fig. 10. The response and recovery time of Ni-doped ZnO film is much faster than of undoped ZnO measured under the same condition reported in our previous work[27]. The crystallite size could affect the sensing characteristics[28]

3.5.2.Sensitivity

The response of the Ni (0.0002M) doped ZnO(0.1M) film, which is tested at different ammonia concentrations at room temperature are reported in fig.11 and the values are obtained by using the following relation

$$S = \frac{Ro - Rg}{Rg} \times 100\% \text{ ---------------------(10)}$$

Doping ZnO with (Ni at 0.0002M) allows a remarkable achievement of the sensing characteristics towards NH_3. The high response of the film can be observed upon exposure to NH_3 concentration as high as 80 ppm. The saturation response value was obtained at higher concentrations (above (120 ppm). The characterization analysis (XRD) indicates the structural quality of the doped sample shows is better than the pure sample. On this basis, we can conclude the less doping (Ni(0.0002M) ZnO(0.1M)) of the film may be the key factor in the enhancement of sensitivity , observed in the present work[29]. The response of doped ZnO film is found to be high with a value 27.33 at 80 ppm of ammonia, compared to the other reported work for sensing ammonia[30] in literature. The Ni ions influence the reaction between the adsorbed ions and NH_3 molecule, which greatly enhances the NH_3 detection ability of Ni-doped ZnO film. This may be due to the effect of nanoparticles and porous morphology as shown in (SEM image).[31]

CONCLUSION

In summary, pure and Ni doped ZnO films with various doping concentrations were are prepared by spray pyrolysis technique. XRD studies revealed that both the films indexed at maximum (101) peak confirm that all films have hexagonal wurtzite structure with poly crystalline nature. Both the films exhibited good transparency in the visible region. The optical band gap of the doped films was less than that obtained for pure film, the lowest value(3.27eV) being observed for 0.0002M doping. From the morphological and topographical characterizations, it is seen that the particles are arranged closely, agglomerate in some places and also are porous in nature, which is suitable for vapour sensor application. Doping ZnO with Ni (at 0.0002M) allows a remarkable application in its sensing characteristics towards NH_3. The high response of the film is measured upon exposure to NH_3 concentration as high as 80 ppm. The quick response and fast recovery time was recorded for 80 ppm at room temperature.

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APPENDIX

Table – 1: *Variation of Texture Co-efficient with Undoped and Ni Doped Films*

S. No	Solution concentration	TC(101)	TC(002)	TC (100)
1	ZnO	1.13	1.04	0.81
2	NiZO-1 (0.0001M)	1.51	1.10	0.34
3	NiZO-2 (0.0002M)	1.69	1.28	0.90
4	NiZO-3 (0.0003M)	1.80	1.30	1.01

Table – 2: *Variation of Lattice Parameters, Strain%, Stress and Crystallite Size as a Function of Doping Concentration*

S. No	Solution concentration	Stress Gpa	Strain%	Lattice Parameter(a) (Å)	Lattice Parameter(c) (Å)	Crystallite size D (nm)
1	ZnO	-1.757	7.561	3.254	5.638	15.72
2	NiZO-1	1.174	-2.560	2.853	4.942	63.87

	(0.0001M)					
3	NiZO-2 (0.0002M)	-5.345	-4.045	2.856	5.135	75.34
4	NiZO-3 (0.0003M)	-3.522	-3.576	2.956	5.156	95.60

Table – 3: *Sensing Parameters with Various Vapour Concentrations*

Ammonia vapour concentration (ppm)	**Response time (sec)**	**Recovery time (sec)**	**Change in resistance when exposed to Air and ammonia ×$10^8\,\Omega$**	**Sensitivity %**
20	20	12	0.4	0.473
40	19	15	1.74	18
80	12	21	2.60	27.33
100	10	39	2.80	24.15
120	9	39	2.30	24.18
140	9	40	2.30	24.18

Figure – 1: *XRD pattern of un doped and Ni doped ZnO thin films obtained from different precursor solution concentrations*

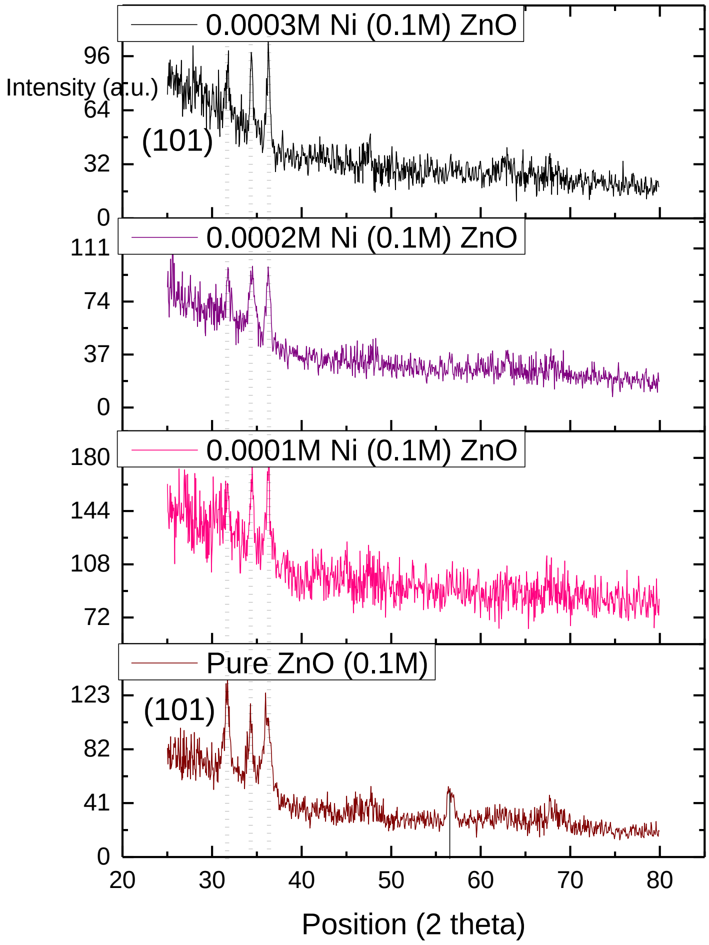

Figure – 2: *Optical Absorbance Spectra of Un Doped and Ni Doped ZnO Thin Films Deposited at Various Concentrations as a Function of Wavelength*

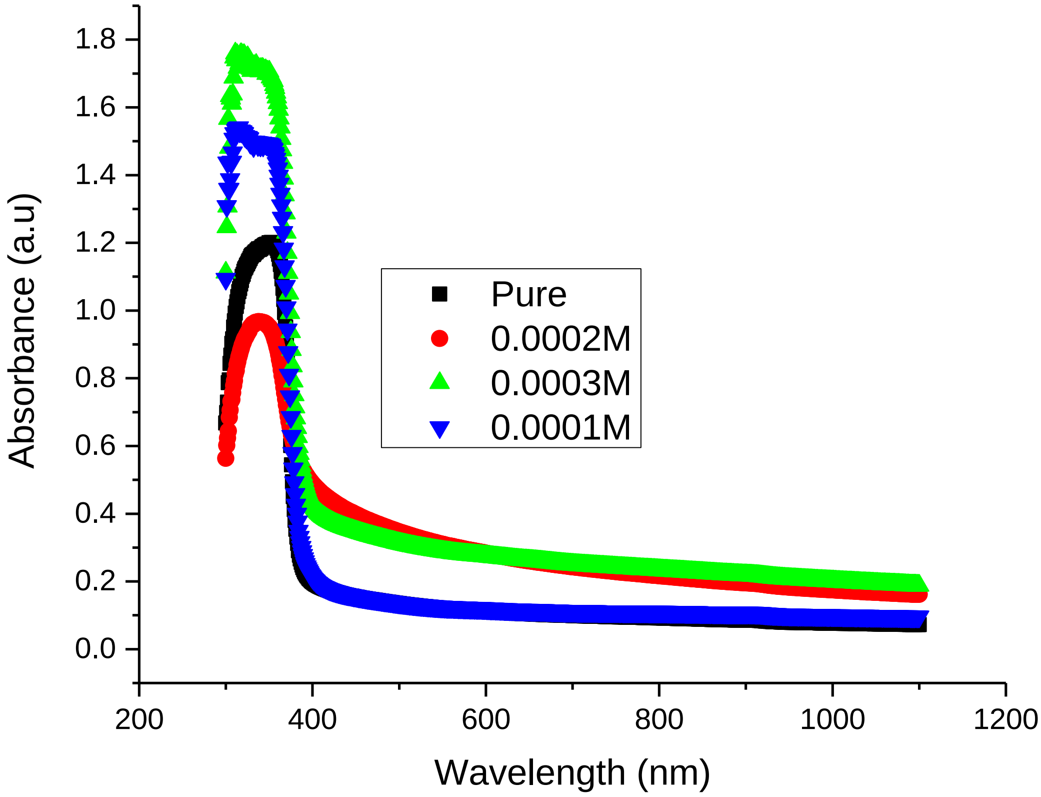

Figure – 3: *Optical Transmission Spectra of Un Doped and Ni Doped ZnO Thin Films Deposited at Various Concentrations as a Function of Wavelength*

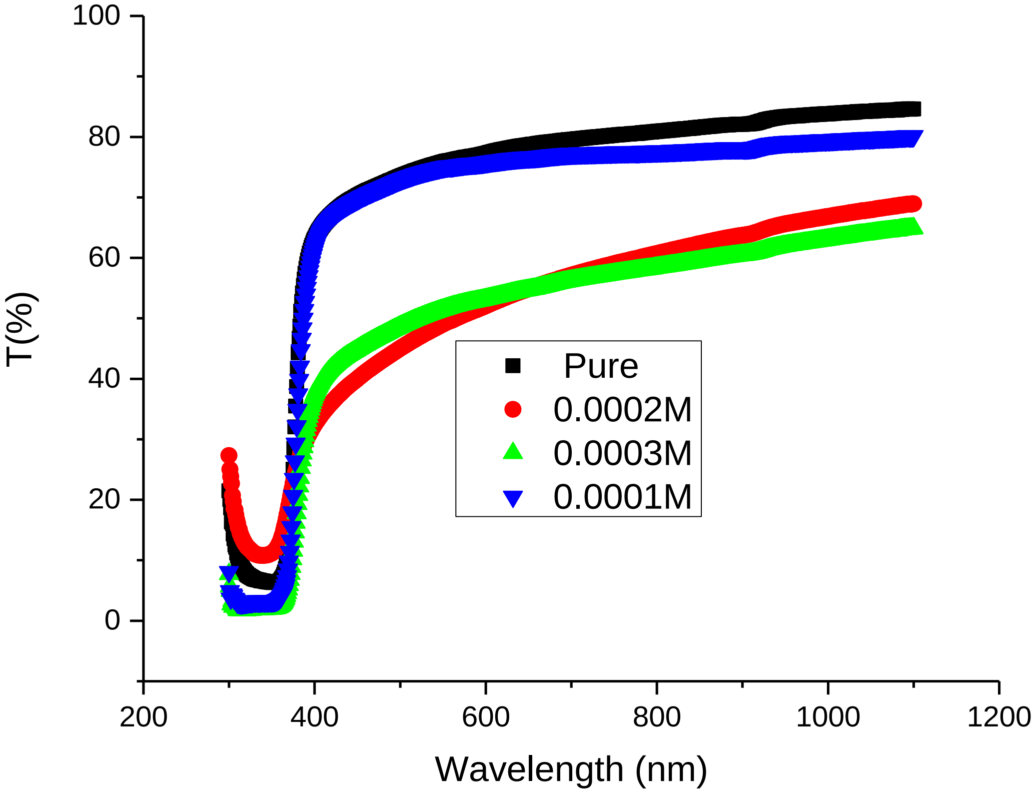

Figure – 4: *Estimates of the Optical Band Gap Energy (Eg) of Un Doped and Ni Doped ZnO Thin Films Using Tauc's Plot as a Function of Concentration*

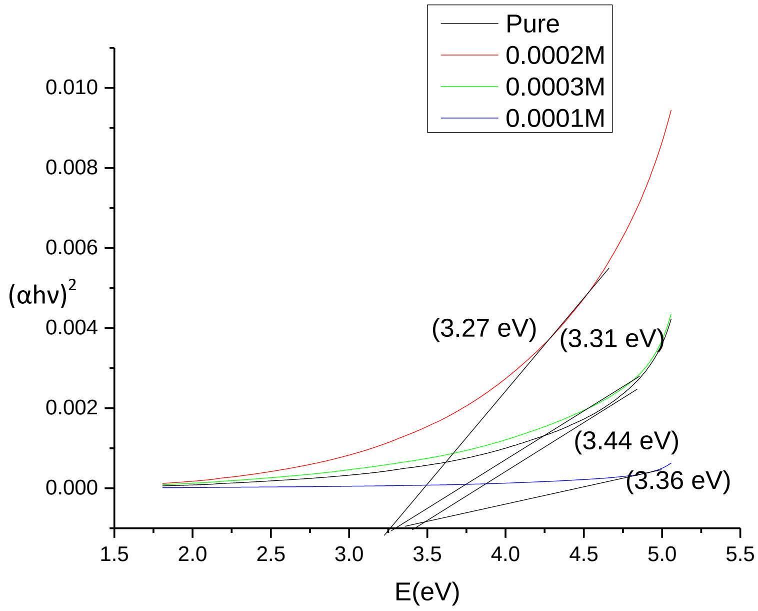

Figure – 5 *(a) and (b) Surface Topography Variation of Un Doped and Ni (0.0002M) Doped ZnO (0.1M) Thin Films 3D Image Over a Scale of 5µm× 5µm*

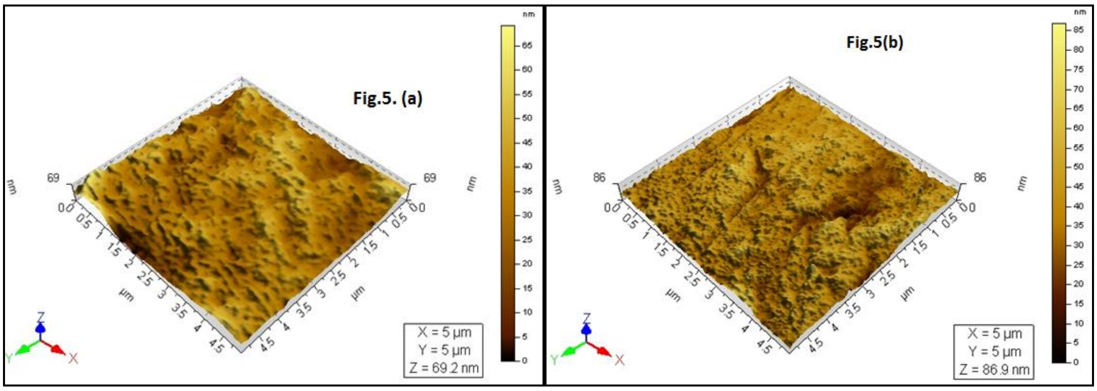

Figure – 6: *(a) and (b) FESEM Images of Un Doped and Ni(0.0002M) ZnO (0.1M) Films*

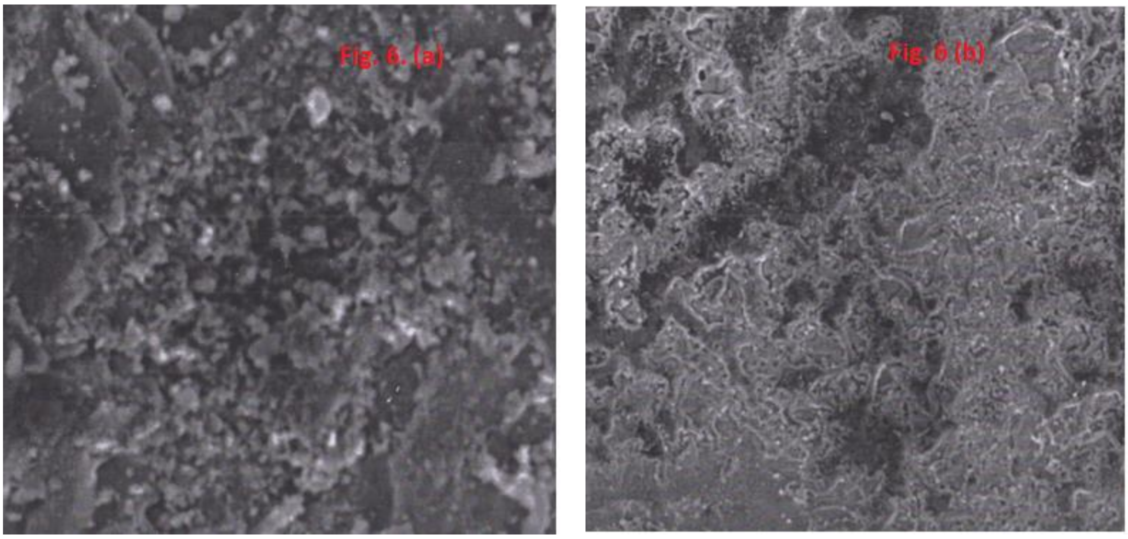

Figure – 7: *Schematic Diagram of the Possible Sensing Process of ZnO Sensor*

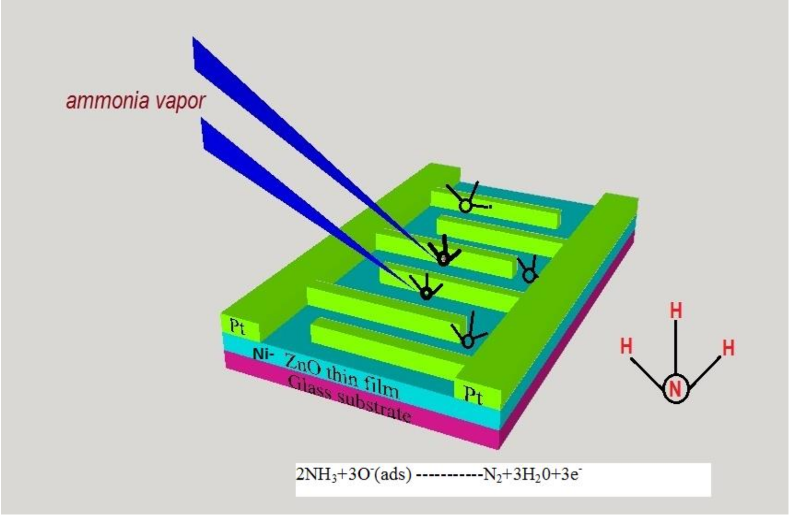

Figure – 8: *Transient Resistance Response of Various Ammonia Vapour Concentration*

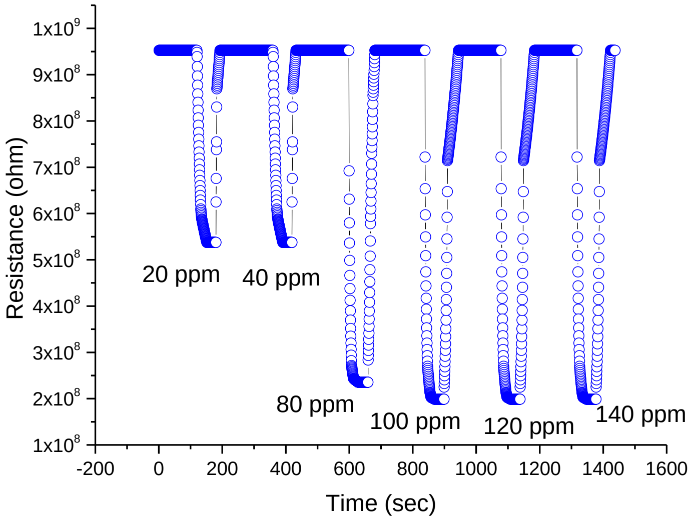

Figure – 9: *Plot of Response and Recovery Times of the Sensor with Varying Vapour Concentrations*

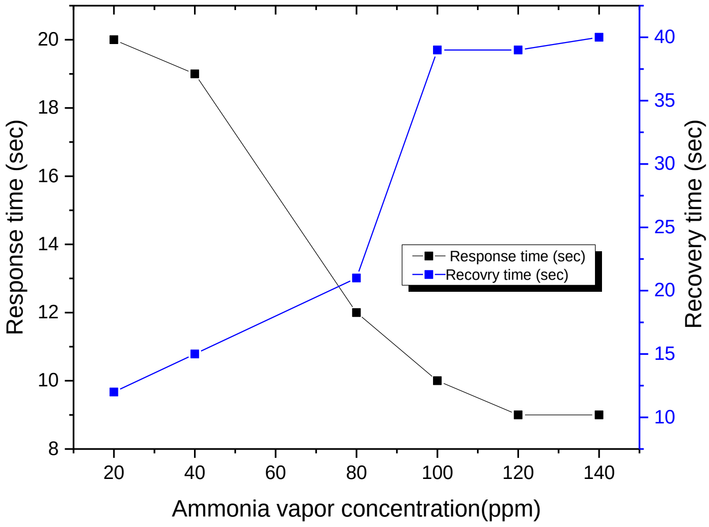

Figure – 10: *Sensing Behaviour of the Film at 80 ppm of* NH_3 *Vapor*

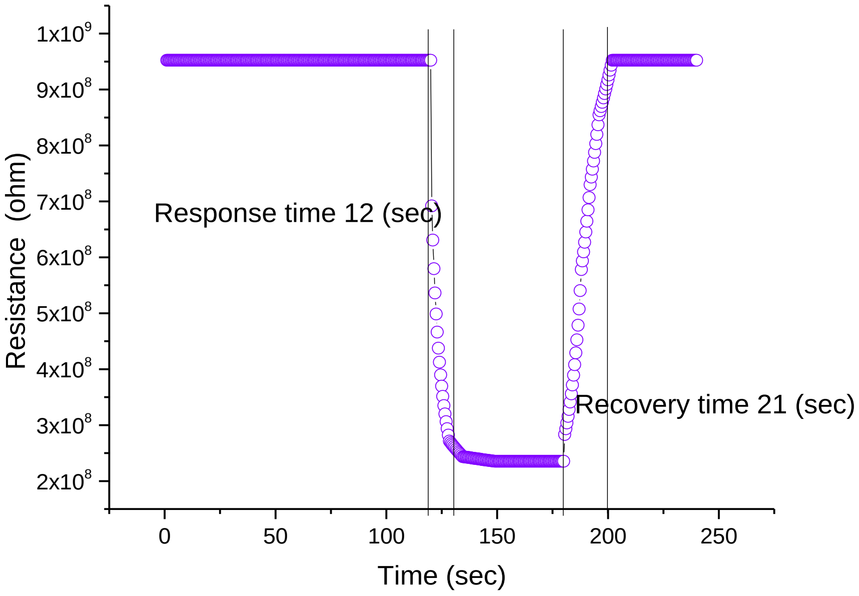

Figure – 11: *Sensing Response of Various Ammonia Vapour Concentrations*